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Review

Thin-layer chromatography in food and agricultural analysis

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Abstract

The TLC analysis of agricultural products, foods, beverages, and plant constituents is reviewed for the period from mid-1995 to mid-1999. Techniques and applications for a wide range of analyte and sample matrix types are covered, with specification of the particular layers, mobile phases, detection methods, and quantification conditions in many cases.

Keywords: Reviews; Thin-layer chromatography; Food analysis; Pesticides

Contents

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1. Introduction

Thin-layer chromatography (TLC) is widely used in laboratories throughout the world for food analysis and quality control. Numerous applications of TLC have been reported in the areas of food composition, intentional additives, adulterants, contaminants, and decomposition involving determinations of compound classes such as amino acids (protein quality), lipids and fatty acids (quality and adulteration of fat), sugars (beverage quality), biogenic amines (storage stability), vitamins (added as nutrients, colorants, and antioxidants), and organic acids (preservatives). TLC has also been widely used to analyze agricultural products and plants. Earlier papers [1–3] and a book chapter [4] reviewed the field of agricultural and food analysis by TLC through mid-1995, and this paper updates coverage to papers published or abstracted from 1995 through mid-1999. References describing advances in techniques and applications to important analyte and sample classes that were published in this period are presented, followed by brief descriptions of selected analyses as examples of typical protocols for food and agricultural TLC analysis.

Modern high-performance TLC (HPTLC) is an efficient, instrumentalized, quantitative method that is carried out on layers composed of particles with a smaller particle diameter, 5 μm compared to 12–20 μm for conventional TLC. Particle size distribution is narrower, layers are thinner, and the development distance is shorter for HPTLC, leading to greater separation efficiency, faster separations, and improved detection limits. Quantitative HPTLC using a densitometric scanner can produce results that are comparable with gas chromatography (GC) and column liquid chromatography (HPLC) when optimally performed. Although TLC, GC, and HPLC are highly complementary, TLC has advantages in many analyses, including the following: simplicity of operation; the availability of many sensitive and selective reagents for detection and confirmation without interference of the mobile phase; the ability to repeat detection and quantification at any time with changed parameters because fractions representing the entire sample are stored on the plate; in-system calibration for quantitative analysis; and cost effectiveness because many samples can be analyzed on a single plate with low solvent usage. A study [5] comparing the cost of a multiresidue HPLC method for determining sulfonamides in meat products with the use of initial TLC sample screening followed by HPLC analysis of suspect samples found that positive samples were identified and confirmed by the latter approach for 20% of the cost of the HPLC multiresidue method and that the combined TLC–HPLC method provides double confirmation of contaminant identity, thereby reducing the possibility of misidentification. Details of the principles

and practice of TLC, comparisons of TLC to HPTLC, applications of these methods to all compound types, and comparisons to HPLC and GC are contained in two books [6,7].

2. Techniques

2.1. Sample preparation and application

Procedures for obtaining, storing, and preparing samples are generally similar to those for GC and HPLC, except sample preparation can often be simplified because the layer is used only once. Some samples can be directly spotted or require only dilution prior to TLC. Solvent extraction (separatory funnel, Soxhlet, and extraction under reflux), macro and micro steam distillation, liquid–liquid partitioning with controlled pH, and liquid–solid column chromatography continue to be used widely for recovery, concentration, and cleanup of samples. However, these traditional approaches are increasingly being replaced by methods such as supercritical fluid extraction (SFE) and solid-phase extraction (SPE) with a cartridge, minicolumn, or disk. The ability to reduce required extract purification in many TLC analyses enables high sample throughput, e.g., in the qualitative and quantitative evaluation of *Passiflora coerulea* flavonoids by HPTLC with detection using two fluorescence reagents for 'fingerprinting' of different representatives of this genera [8].

Examples of SFE for recovery of analytes prior to TLC include the determination of semivolatile compounds in cinnamon and cassia [9], cyanazine herbicides in soil [10], study of the composition of ginger essential oil [11], and screening of soil for residues of 20 pesticides of multiple classes [12].

SPE was used for recovery of benzoic and sorbic acid preservatives from beverages [13] and cholesterol oxidation products from foods [14] prior to TLC analysis. SPE on aminopropyl cartridges was used to recover 28 polar pesticide residues from crops [15].

In general, samples seldom require derivatization prior to TLC analysis, as is often the case with HPLC and, especially, GC. Hydrolysis of the sample by acid, base, or enzymes may be required for specific analyses, such as amino acids in proteins or sugars in starch.

Sample preparation methods prior to TLC analysis are specified in the applications described in Section 4 and a few of those in Section 3.

Nanoliter to microliter volumes of standard and sample solutions are applied manually to the layer as spots or bands with a fixed- or variable-volume micropipette or with an instrument. Instrumental, automated sample application is critical for accurate and precise quantitative analysis involving densitometry.

2.2. Layers and mobile phases

Many types of adsorption, partition, and ion-exchange layers have been used for food analysis, but the great majority of all separations are carried out currently on normal- or straight-phase (NP) precoated silica gel TLC or HPTLC layers as seen in Sections 3 and 4. Plates containing a preadsorbent or concentrating zone facilitate rapid sample application and formation of compact, band-shaped zones after development. Other NP layers that are used for some food analyses include alumina and cellulose. Reversed phase (RP) TLC is performed mostly on chemically bonded C-18, C-2, C-8, and diphenyl layers.

The use of corn starch, rice starch, talc, and impregnated corn starch layers was reported for densitometric quantification of organic acids, carbohydrates, fat-soluble vitamins, amino acids, and anthocyanins in foods [16]. Hydrophilic diol-bonded layers were used for identification (preparative TLC) and densitometry of 2-hydroxycinnamaldehyde in commercial cinnamons [17] and for densitometry of sugars in foods after automated multiple development (AMD) [18].

Impregnation of plates with a reagent may allow improved resolution for certain compound classes. For example, TLC-densitometry on silica gel layers impregnated with silver ions (argentation TLC) provided determination of petroselinic, *cis*-vaccenic, and oleic acid phenacyl esters in some seed oils of the Umbelliferae [19], fatty acid isomers in commer-

cial spreads [20], and triacylglycerols in milk fat [21].

For best results, plates are usually purified by washing with a single solvent or solvent mixture before use. Ascending development with dichloromethane–methanol (1:1) has been widely used for this purpose. However, dipping in methanol has been shown to be more effective than predevelopment for layer cleaning [22].

The mobile phase for TLC is usually chosen by trial and error guided by literature descriptions of earlier similar separations or by use of systematic mobile phase design and optimization models. The most widely used of these is the 'Prisma' model, which was used to select the optimum mobile phase for the overpressured layer chromatography (OPLC) analysis of food antioxidants [23]. Mobile phases for a variety of analyte classes are specified in many of the TLC applications described in Sections 3 and 4.

2.3. Development of the layer

TLC is usually performed in one-dimension by gravity-flow ascending development with a single mobile phase in a solvent vapor-saturated, paper-lined glass N-chamber. Multiple development in one direction with the one or more mobile phases can improve resolution in some cases. For example, the cyanogenic glycoside linamarin was quantified in Cassava by HPTLC on silica gel by double development with ethyl acetate–acetone–water (4:5:1) for 3 cm and then ethyl acetate–formic acid–water (6:1:1) for 8.5 cm, followed by detection with aniline–diphenylamine–phosphoric acid reagent and densitometry at 525 nm [24]. Multistep, manual gradient elution in a sandwich chamber was used for the separation and densitometry of the colored pigments in red wine on reversed-phase paraffin-impregnated silica gel plates [25].

AMD is becoming widely used for high-efficiency automated HPTLC separations prior to quantification by densitometry. In AMD, an automated instrument produces a 20–25 step mobile phase gradient ranging from stronger to weaker elution power, with successive developments occurring over a longer incremental distance (typically 3 mm or less). Large spot capacities result because of the reconcentration effect caused by the multiple development, as well as separation of complex mixtures with a wide range of polarities because of gradient development. An example of an application is the separation and analysis of sugars in foods on diol-bonded layers with a 15-step acetonitrile–water gradient decreasing linearly from 35 to 15% water [18].

Two-dimensional (2D) development improves resolution of a complex mixture by development with two mobile phases at right angles. As an example, the effect of soybean pretreatment on the phospholipid content in crude and degummed oils was studied by qualitative 2D TLC on silica gel with chloroform–methanol–28% ammonia (13:5:1) in the first direction and chloroform–methanol–acetic acid–water (170:25:25:6) in the second [26].

Forced flow development by OPLC involves pumping the mobile phase through a layer that is sandwiched between a rigid plate and a flexible membrane under pressure. OPLC results in large spot capacities and short development times, and gradient elution can be used. OPLC with dichloromethane–ethyl acetate (93:7) mobile phase and densitometry at 313 nm has been used to determine biogenic amines in cheese [27,28] and to study the formaldehyde cycle in budding and apoptosis of trees [29]. Aflatoxins extracted from corn were determined without cleanup by 2D OPLC using ether–chloroform and chloroform–ethyl acetate mixture in the first direction and chloroform–ethy1 acetate–tetrahydrofuran (THF) (10:15:1) in the second direction, followed by fluorescence densitometry at 365 nm/380 nm [30]. Silica gel HPTLC–OPLC with overrun and stepwise gradient elution and densitometry at 313 nm was used to separate and quantify seven dansylated biogenic amines in vegetables [31].

2.4. Detection of zones

Many hundreds of chromogenic and fluorogenic reagents are described in the literature (e.g. Ref. [7]) for detection on layers of analytes that cannot be visualized by natural color in daylight or by natural fluorescence or fluorescence quenching under 254 or 366 nm UV light. Fluorescence detection is preferred when possible because of its specificity and sensitivity. An example is the detection of nitroimidazoles from poultry on silica gel HPTLC plates by reduction of the nitro group followed by reaction with

fluorescamine fluorogenic reagent (detection limit 15–20 ng) or by spraying with pyridine (3–25 ng) and viewing under 366 nm UV light [32]. Although generally less sensitive, chromogenic reagents are widely used, e.g., for detection of monosodium glutamate in foods prior to quantification by densitometry at 520 nm [33]. Detection methods and reagents are specified in many of the applications described in Sections 3 and 4 below.

New detection methods are reported regularly in the TLC literature and are reviewed biennially by Sherma [34]. As an example, a new method for detection of animal neutral lipids prior to quantification by densitometry at 430 nm involved spraying with three different reagents: (1) 3% cupric acetate in 6% aq. phosphoric acid solution, followed by heating at 100°C for 10 min; (2) 35% aq. sulfuric acid, followed by heating at 140°C for 10 min; (3) procedure (1) followed by procedure (2) [35]. The specific detection of phosphorothionate and phosphorothiolothionate pesticides extracted from mushrooms as deep magenta zones by reaction with 4-amino-*N*,*N*-diethylaniline is a second new method; the limit of detection is 0.05–0.5 μg/zone [36].

2.5. Documentation of chromatograms

Photography of chromatograms is an important method for recording and preserving the results of a TLC analysis; such documentation is becoming increasingly important to meet GLP (good laboratory practice) and GMP (good manufacturing practice) requirements. The optimum conditions to be used for photodocumentation of chromatograms were described and examples were presented involving colored and monochrome photography of colored, fluorescent, and fluorescence-quenched zones, including lipids, aspartame, and quinine, on silica gel plates [37]. Photographic documentation is currently being replaced at an increasing rate by video camera and computer scanning technology.

2.6. Quantification by densitometry

Quantitative analysis is carried out in situ by measurement of sample and standard zones on layers with a slit-scanning densitometer or video or CCD camera (image processing). Slit-scanning instruments have a number of advantages and were used in most of the densitometry papers cited in this review. However, more papers are being published each year with quantification based on image analysis. For example, plant phenolics were quantified on normal- and reversed-phase layers using video-densitometry, and results were compared with those from a dual-wavelength, flying spot UV densitometer [38]. Densitometry is best performed at the wavelength of maximum absorption for visible/UV absorption scanning and the maximum excitation/emission wavelengths for fluorescence scanning; these optimum measurement wavelengths are specified in some of the applications in Sections 3 and 4 below.

As mentioned above, instrumental initial zone application is necessary to achieve accurate and precise quantitative results. As an example, a nebulizer-based sample application system was described for the quantitative analysis of aflatoxins in foods in the low picogram range utilizing a CCD camera [39]. Use of a CCD camera or densitometer was compared for quantification of saccharose in fermented and non-fermented soybean flour in reflectance and transmission modes on a silica gel layer developed with acetonitrile–water–methanol (68:12:1) with detection using diphenylamine–aniline chromogenic reagent [40].

Validation of results to meet GLP/GMP standards is increasingly important. Complete validation of an HPTLC method for determination of caffeine in cola according to ICH guidelines for selectivity, analyte stability, linearity, precision, and robustness was demonstrated for quantification with a slit-scanning densitometer and an image-analyzing system [41].

2.7. Special techniques

Preparative layer chromatography (PLC) is carried out on thicker layers with application of larger weights and volumes of samples in order to separate and recover 10–1000 mg of compound for further analysis. As an example, new colored compounds from the Maillard reaction between xylose and lysine were isolated by PLC on silica gel layers with a concentrating zone using chloroform–ethy1 acetate–ethanol (15:2:2) and methyl acetate–water (8:1) mobile phases, detection in daylight and under 366 nm UV light, and quantification by HPLC with a

diode array detector after elution from the layer [42]. The preparative TLC of vanillin on silica gel with dichloromethane–ethanol (19:1) and detection under 254 nm UV light was part of a study of authenticity assessment of vanillas [43].

The separation and measurement of radio-labelled compounds on TLC plates is a widely used method for following metabolism reactions of compounds such as drugs and pesticides. Radio-TLC with a scintillation counter was used to study inhibition of aggregation and alteration of eicosanoid metabolism in human blood platelets caused by curcumin, a major component of food spice tumeric [44]. Soil-bound residues of the pesticide cyprodinil and their plant uptake was studied by silica gel TLC with toluene–methanol (9:1) or chloroform–ethanol–acetic acid (90:10:1) and detection by radioscanning [45]. New fluorinated fungicides and metabolites were detected in plant cells by TLC on silica gel by 2D TLC developed with toluene–ethanol (4:1) and then ethyl acetate at right angles, followed by ^{19}F-NMR or ^{14}C-label autoradiography [46], and autoradiography was also used to study the degradation of four commonly used pesticides including 2,4-D in Malaysian agricultural soils after TLC on silica gel developed with benzene–hexane–acetone (25:25:1) [47].

On-line coupling of reversed-phase HPLC and normal-phase TLC provides a multi-mechanism analytical system with high peak capacity. An on-line system consisting of a microbore HPLC syringe pump and a modified TLC personal computer autosamp1er enabling effluent to be sprayed from the column in fractions onto the plate at defined locations was applied to the densitometric determination of multiple pesticide residues [48] and iprodion residues with AMD [49] in foods.

3. Applications

3.1. Alkaloids

Lupanine and 3β-hydroxylupanine were determined in agricultural products by silica gel TLC with cyclohexane–diethylamine (7:3), visualization with Dragendorff reagent, and NMR analysis [50]. In a study of heat-induced damage in potato tubers, the glycoalkaloids solanidine, α-solanine, and α-chaconine were separated on cellulose in chloroform–methanol–3% ammonia (50:50:3), detected by exposure to iodine vapor, and determined by photometry after elution [51]. The occurrence of the tobacco alkaloid myosmine in nuts and nut products was investigated using analytical and preparative TLC on silica gel with chloroform–methanol (9:1), detection under UV light, and HPLC or GC–MS analysis [52]. Silica gel TLC with chloroform–methanol–1% ammonia (2:2:1, bottom layer) mobile phase and detection using ethanolic sulfuric acid reagent was used in the study of the tomato glycoalkaloid tomatidenol-3β-lycotetraose [53]. Purine alkaloids (caffeine, theobromine, theophylline) were determined simultaneously in daily foods and health drinks by Extrelut column extraction and silica gel HPTLC with dichloromethane–ethanol–28% ammonia (180:17:3) and quantification by densitometry at 275 nm with *p*-hydroxybenzaldehyde as the internal standard; less than 3 h was required for complete analysis of ten samples [54].

3.2. Amides, amines, and amino acids

HPTLC of ANOT (2-amino-5-nitro-*o*-toluamide) and primary metabolites on silica gel with chloroform–ethyl acetate–methanol (5:5:1) and detection under 366 nm UV light and by exposure to nitrous acid vapors and spraying with Bratton–Marshall reagent was used to study the chemical reduction of zoalene to ANOT for use in zoalene residue analysis [55]. Oryzalin (3,5-dinitro-N^4,N^4-dipropylsulfanilamide) and degradation products were separated on silica gel with a variety of mobile phases and detected and quantified by densitometry in studies of sorption and degradation in agricultural products [56].

Resveratrol and biogenic amines were determined in red wine by silica gel TLC with ethyl acetate–heptane (9:1) and ethyl acetate–methanol–ammonia (4:3:1), respectively, and fluorescence densitometry of resveratrol directly and of dansyl derivatives of the amines [57]. Biogenic amines were extracted from foods with 5% trichloroacetic acid, the extract washed with diethyl ether, and dansyl derivatives were prepared, separated by multiple development TLC, and quantified by densitometry at 254 nm; the

limits of detection were 5–10 ng, RSD 0.4–6.4%, and recoveries 86–93% [58].

Changes in the amino acid composition of dehydrated orange juice during non-enzymatic browning were detected by qualitative identification on cellulose with 1-butanol–pyridine–water (2:3:1) mobile phase and detection by spraying with 2,3,5-tripheny1-2*H*-tetrazolium chloride–NaOH reagent [59]. Tryptophan and related metabolites were determined in foods by TLC on cellulose with chloroform–methanol–ammonia (12:7:1) and densitometry with a fiber optic remote sensor fluorescence instrument at 280 nm/>347 nm [60]. Taurine was determined in energy drinks by semiquantitative TLC based on the color formed with ninhydrin detection reagent [61].

3.3. Anthocyanidines

Petunidin 3-*O*-α-rhamnopyranoside-5-*O*-β-glucopyranoside and other anthocyanidines from flowers of *Vicia villosa* were identified by cellulose TLC using 1-butanol–acetic acid–water (4:1:5) and formic acid–HCl–water (1:1:2) as mobile phases [62].

3.4. Anthraquinones

TLC on silica gel with chloroform–light petroleum (1:1), chloroform–ethyl acetate (1:1), and benzene–light petroleum–ethyl acetate (1:2:1), detection under 254 and 366 nm UV light, and identification based on Vis–UV spectra was used to study the chemotaxonomic significance of anthraquinones in the roots of asphodeloideae (Asphodelaceae) [63].

3.5. Antibiotics

Separation on silica gel with chloroform–methanol (10:1), detection under UV light, and microbiological quantification after elution by an agar diffusion method was used to analyze oxytetracycline and chlorotetracycline in animal feeds in the presence of 11 other drugs: three nitrofurans, two macrolide antibiotics, three sulfonamides, two coccidiostatics, and one antibacterial growth promoter [64]. Similar methods were used to quantify erythromycin and tylosine in animal feeds in the presence of two tetracycline antibiotics and the other drugs listed above, except that the mobile phase contained 0.04 parts by volume of ammonium hydroxide [65]. Residues of the quinolonic antibiotics oxolinic acid and flumequine were quantified in fish tissue by densitometry at 320 nm with nalidixic acid internal standard, after extraction with a 7:3 mixture of acetonitrile and an aqueous solution of KCl and KOH, by HPTLC on silica gel layers impregnated with dipotassium hydrogenphosphate and developed with toluene–ethyl acetate–90% formic acid (6:3:1), with detection using sulfuric acid–hydrochloric acid reagent; recovery was >97%, RSD 1–2%, and sensitivity 10 ppb [66].

3.6. Carbohydrates

In a study of African mango glycosidically bound volatile compounds, saccharides and aglycones were separated on silica gel with ethyl acetate–2-propanol–water (13:6:3) and detected with *N*-(l-naphthyl)ethylenediamine dihydrochloride reagent [67]. Thermolysates of sugars (1,6-anhydroglucose, D-glucose, dextrin, and starch) were analyzed by HPTLC on silica gel with a 23-step AMD based on chloroform–methanol–water, detection with aniline–diphenylamine–phosphoric acid reagent, and densitometry at 385 nm [68]. Hydrolysis products of glycosides (glucose, rhamnose, rutinose, pNP-xyloside, eugenylprimeveroside) were qualitatively identified by separation on silica gel with ethyl acetate–2-propanol–water (65:30:10) and detection with napthorescorcinol–ethanolic sulfuric acid reagent in a study of an endoglycosidase from grape skin hydrolyzing potentially aromatic disaccharide glycosides [69]. Quantitative monitoring of maltooligosaccharides and monosaccharides in beers on amino-bonded HPTLC layers by gradient elution-AMD was found to be important for controlling brewing processes and characterization of final product taste [70]. Analysis of beet and cane molasses in the sugar industry by HPTLC–AMD and –OPLC was described in detail [71].

3.7. Flavonoids

Separation of HCl vapor-hydrolyzed flavonoid glycosides from endophyte-infected blue grass (*Poa ampla*) (tricin, isoorientin, and related compounds) was performed on a silica gel plate by development

with chloroform–methanol (10:1) and with the lower layer of chloroform–methanol–water (7:3:1); the aglycons and sugars were detected with ethanolic sulfuric acid [72]. The distribution of flavonoids, alkaloids, acetophenones, and phoroglucinols in *Bosistva floydii* and *B. medicinalis* was studied using TLC on silica gel developed with hexane–ethy1 acetate (1:1) [73], and flavonoids in extract of artichoke were identified by development with ethyl acetate–formic acid–water (8:1:1) [74]. Buckwheat was analyzed for antioxidant flavonols and proanthocyanadins using TLC on cellulose with butanol–acetic acid–water (4:1:5) and with 30% acetic acid; detection was by viewing under UV light, ammonia vapor, and aluminum chloride reagent [75]. Flavonol glycosides from the seed coat of a new manteca-type dry bean (*Phaseolus vulgaris* L.) were separated on silica gel with chloroform–methanol (4:1 and 1:1) and chloroform–methanol–water (8:2:1) and detected by spraying with 20% sulfuric, heating, and viewing under UV light, while 11 proanthocyanidines were chromatographed on cellulose with butanol–acetic acid–water (4:1:5) and detected by spraying with 5% vanillin in ethanol–conc. HCl (4:1) [76]. Variety differentiations and authentication of blood orange juice was based on flavanone glycoside content, which was determined by PLC on C_{18} bonded silica gel developed first with water–acetonitrile–THF–acetic acid (85:12:12:1) and then (81:14:2:3), detection under UV light, extraction from the layer, and NMR analysis and hydrolysis [77]. Silica gel TLC with water–methanol–ethy1 acetate (3:4:2) and photometry after elution was used to study the tissue and spatial distribution of flavanol glycosides and peroxidase in onion bulbs and stability of the glycosides during boiling of the scales [78]. Citrus polymethyloxylated flavones (sinensetin, nobiletin, tangeretin, heptamethoxyflavone, quercetogetin) were found to confer resistance against *Phytophthora citrophthera*, *Penicillum digitatum*, and *Geotrichum* species based on silica gel TLC with benzene–acetone (1:1), detection under UV light, and identification by photometry and MS [79]. Polymethoxylated flavones (sinensetin, nobiiletin, tangeritin, tetra-*O*-methylisoscutellarein, tetra-*O*-methylscutellarein, isoinensetin) isolated from cold pressed tangerine peel oil solids were analyzed by HPTLC on silica gel with chloroform–methanol (9:1) mobile phase and on C_{18} silica gel with methanol–water (4:1), ethanol–water (3:2), and acetonitrile–THF (9:1) and detection on both layers under UV light and with ethanolic sulfuric charring reagent on silica gel [80]. Flavones and flavone glycosides from biflavones in vegetable extracts were separated by PLC on silica gel and C_{18} plates and identified by fast atom bombardment (FAB) MS and UV spectrometry of the isolated compounds, simultaneous analytical TLC of sample and reference compounds, and acid hydrolysis performed directly on the layer [81].

3.8. Imidazoles

Residues of three nitroimidazoles (ronidazole, dimetridazole, and their major metabolite hydroxydimetridazole) were qualitatively determined at 2–5 μg/kg in pork and poultry muscle tissue by extraction, SPE cleanup, and silica gel HPTLC by use of multiple development with methanol and ethyl acetate and detection by spraying with pyridine and observation under 312 nm UV light [82].

3.9. Lipids

Lipid classes including fatty acids and triglycerides were quantified in papaya seed oil by silica gel PLC, analytical argentation TLC, detection with bromine and sulfuryl chloride vapors, and densitometry at 450 nm [83].

Sterols in consumable fats were determined as a group by GC–MS and TLC on silica gel, silica gel–Kieselguhr, alumina, and C_{18} layers developed in a DS-type sandwich chamber and by OPLC, with detection by use of phosphomolybdic acid (PMA) and modified Liebermann–Burchard reagents [84]. 4,4-Desmethylsterols and triterpene diols extracted with diethyl ether from alkaline-hydrolyzed plant oils were separated on silica gel with preadsorbent zone using light petroleum–diethyl ether (7:3) mobile phase, detected by spraying with 0.2% ethanolic 2,7-dichlorofluorescein, and identified and quantified by GC–MS [85].

Milk fat triglycerides were identified by TLC on silica gel impregnated with silver nitrate and developed with chloroform; compounds were detected by spraying with 0.15% ethanolic 2′,7′-dichloro-

fluorescein and observation under UV light, eluted, and quantified by capillary GC [86]. Mono-, di- and triacylglycerides were analyzed in vegetable oils degraded during frying by separation on C_{18} layers with dichloromethane–ethyl acetate–methanol–acetic acid (27:22:38:13), detection at 0.4 μg levels with PMA reagent, and densitometry [87].

The fatty acid ester content and their antioxidant activity in paprika were studied using silica gel TLC with acetone–light petroleum (15:85) mobile phase [88]. Vernolic and cyclopropenoic fatty acids in *Piper nigrum* seed oil were separated by analytical TLC on silica gel with diethyl ether–hexane (2:8 and 3:7) and detected by spraying with dichlorofluorescein and viewing under UV light; in addition, PLC was carried out to separate mixed fatty acids into oxygenated and non-oxygenated fractions [89]. Fatty acid methyl esters in edible fats were isolated by PLC on silver nitrate- impregnated silica gel and quantified by GC after elution with diethyl ether [90], and they were identified in *Cannobis sativa* and *Sorghum bicolor* oils by silica gel TLC with diethyl ether–hexane (1:9) mobile phase [91]. An official method for determining erucic acid in oils and fats intended for use in food manufacture is based on silica gel TLC with toluene–hexane (9:1) and detection with 2,7-dichlorofluorescein reagent [92].

Mono- and disialogangliosides and 'red protein' from *Scomber scombrus* muscle were identified by development of silica gel layers with 1-propanol–water (7:3) and detection by spraying with resorcinol reagent [93].

Phospholipid hydroperoxides and their parent phospholipids were analyzed as primary oxidation products in cooked turkey meat extracts on HPTLC silica gel developed with hexane–diethyl ether (3:2) to remove the neutral lipids and then chloroform–ethanol–methanol–triethylamine–water (30:25:10:3-5:8) to resolve the phospholipids; detection was by dipping in *N*,*N*-dimethyl-*p*-phenylenediamine–acetic acid–methanol reagent and heating, followed by densitometric quantification at 654 nm [94]. Soybean phospholipids were analyzed by double development of silica gel with chloroform–methanol–acetic acid–methanol–water (35:25:4:14:2.2) and hexane–diethy1 ether (4:1) mobile phases, detection with PMA and Dittmer reagents, and densitometry at 650 nm [95]. A standard German (DGF) HPTLC method was described for separation of all animal and plant lecithins and mixtures or fractions of phospholipids [96].

3.10. Pesticides

General reviews of pesticide TLC, including some information on the analysis of foods, crops, and other agricultural samples, were published earlier [97–99].

Six benzoyl urea insecticides were analyzed in plant food products by TLC with AMD [100]. Degradation products of fungicidal ethylenebisdithiocarbamates on oxidation with potassium permanganate were identified by silica gel TLC with 95% ethanol or chloroform–1-butanol–methanol–water (200:10:2:1) mobile phases and detection under 254 nm UV light or by use of iodine vapor or nitroferricyanide, Ehrlich, or dithizone spray reagents [101]. Eleven rodenticides were quantified in animal feed on silica gel developed with hexane–ethyl acetate (7:3) and on C_{18} bonded silica gel with methanol–water–acetic acid (75:25:0.6) and densitometry at 310 nm [102]. Atrazine, propham, chloropropham, diflubenzuron, α-cypermethrin, and tetramethrin were determined in soil with recoveries of 79–103% by acetone ultrasonic extraction, reversed-phase TLC with a mobile phase chosen by computer-assisted optimization, and slit-scanning densitometry [103].

Pentachlorophenol (PCP) was determined on wood at a level of 100 ppm/100 mg sample by silica gel HPTLC with toluene mobile phase and detection after nitric acid-mediated oxidation of PCP to tetrachloro-1,4-benzoquinone by spraying with acid reagent [104]. An HPTLC method for quantitative determination of propham in the germinator inhibitor Tixit and its validation were described; the method employed silica gel 60 layers developed with dichloromethane, detection under 254 nm UV light (20–30 ng/spot sensitivity), and densitometry at 228 nm [105]. For the study of the microbial transformation of prosulfuron, analytical TLC and PLC were carried out on silica gel with dichloromethane–acetonitrile–formic acid (84:14:0.25 and 75:25:1) and detection under 254 nm UV light followed by consecutive spraying with ceric ammonium sulfate and Dragendorff reagents [106]. Acifluorfen sorption and sorption kinetics were studied by densitometry on HPTLC silica gel layers after develop-

ment with toluene–ethy1 acetate–acetic acid–water (100:100:2:1) [107].

3.11. Phenols

The TLC of glucopyranosyl sinapate and other phenolic compounds obtained from rapeseed using Sephadex LH-20 column chromatography and C_{18} semipreparative HPLC was studied on silica gel, C_{18}, and cellulose layers with 11 different mobile phases; qualitative identification was best obtained using HPTLC silica gel with benzene–methanol–acetic acid (90:16:8) mobile phase and detection by spraying with an aqueous solution of ferric chloride and potassium ferricyanide [108]. Phenolic components extracted from red wine (gallic acid, protocatechulic acid, catechin, chlorogenic acid) were separated from pigments on cellulose with 20% methanol, detected by dipping into 5% ferric chloride, and quantified by densitometry at 700 nm; red wine pigments were quantified on silica gel by densitometry at 540 nm after development with 1-butanol–butyl acetate–formic acid–water (13:5:2:3) mobile phase [109]. Antioxidant catechins from buckwheat groats were identified by TLC on cellulose with 1-butanol–acetic acid–water (4:1:5) and subsequently 30% acetic acid and detection under UV light after treatment with ammonia vapor or spraying with vanillin reagent; lipids were detected on silica gel with hexane–diethyl ether (19:1) and detection by sulfuric acid charring [110]. The phenolic compounds with antioxidative activity present in deodorized rosemary extract used to stabilize butter were identified by TLC on silica gel with chloroform–methanol–water (65:35:10), with detection by spraying with ferric chloride–potassium ferricyanide or a solution of β-carotene and linoleic acid and observing the delay in bleaching of the carotene [111]. Phenols from honeybush tea were identified by analytical TLC and PLC on silica gel using double development with each of two mobile phases, hexane–benzene–acetone–methanol (40:40:15:5) and chloroform–benzene–acetone (50:45:5), and detection with sulfuric acid–formaldehyde (40:1) [112].

Butylated hydroxytoluene (BHT) was determined in gum base with a detection limit of 25 ng by double development on silica gel with hexane (12 cm) and light petroleum (10 cm), detection with 20% ethanolic PMA reagent, and densitometry at 600 nm [113]. 2,3-Hydroxy-1-guaiacylpropan-1-one was identified in brandies by analytical TLC on silica gel and analytical and preparative 2D TLC on cellulose, with detection under UV light and by spraying with diazotized 4-nitroaniline, phloroglucinol, or catechin reagent [114].

3.12. Pigments and dyes

Optimal TLC systems for separation of oxygenated carotenoids [115] and Cu(II) complexes of chlorophylls and allomerization products of pheophytins *a* and *b* [116] were described. Analytical TLC and PLC on silica gel with light petroleum–acetone–diethylamine (10:4:1) and detection under UV light was used to identify the carotenoids capsolutein (as cucurbitaxanthin), zeaxanthin, and lutein from *Capsicum annuum* [117]. TLC of 13 carotenoids on silica gel, MgO–Kieselguhr (1:1), and NaOH-impregnated silica gel plates using light petroleum, diethyl ether, and light petroleum–acetone (9:1) mobile phases and visualization under UV light was carried out to identify the pigments in yellow passion fruit [118]. Capsanthin was quantified in paprika red pigments by TLC on silica gel with light petroleum–benzene (3:1) and hexane–benzene–ethy1 acetate–ethanol (20:2:5:2) and densitometry at 470 nm [119]. TLC and HPLC were used together to detect the carotenoids β-carotene, lycopene, β-cryptoxanthin, rubixanthin, zeaxanthin, and lutein in *Rosa canina* (Rosaceae) fruits [120].

Traces of coal tar dyes in 48 food samples were identified by separation on C_{18} layers developed with acetonitrile or methyl ethyl ketone–methanol–5% aq. sodium sulfate (1:1:1) followed by measurement of visible absorption spectra with sample concentration techniques to improve detection limits 10–20-fold [121]. Quinoline yellow, sunset yellow, cochineal red A, indigo carmine, tartrazine, amaranth, and erythrozine food dyes were separated by OPLC with ammonia–methanol–ethy1 acetate (1:3:6) and ammonia–methyl ethyl ketone–1-butanol (2:3:5) mobile phases and quantified by densitometry [122]. Identification and quantitative analysis of 12 dyes in food extracts were performed by elution from an XAD-2 column with acetone, methanol, and water,

HPTLC on silica gel with 2-propanol-1-propanol-1-butanol–ammonia–water (8:4:4:2:1), and densitometry at the maximum absorption wavelength of each compound; the detection limit was 4–10 ng/zone [123]. A new method was described for quantitative assay of betanine in red beet root dye samples by dual wavelength absorption densitometry on cellulose layers [124]. Synthetic dyes were identified and quantified in wine, wine-containing, and non-alcoholic beverages and spirits at a detection limit of <5 mg/l by silica gel and cellulose TLC and densitometry [125].

3.13. Saponins

Antimicrobial saponins of *Yucca schidigera* were identified by TLC on silica gel with 2-octanol–3-methylbutanol–acetone–methanol–water (3:5:2:1:1) and visualization by spraying with 10% sulfuric acid and heating at 120°C for 20 min [126]. The triterpenoid saponin content of *Saponaria officinalic* cell and *Gypsophila paniculata* root suspension cultures was quantified by HPTLC on silica gel with chloroform–methanol–water (12:9:1) [127]. Alfalfa saponins and their implication in animal nutrition were studied by qualitative 2D TLC for identification with quantitation by densitometry; major biologically active saponins were detected by spraying plates with a suspension of red blood cells [128].

3.14. Sulfonamides

Sulfathiazole, phthalylsulfathiazole, sulfaquinoxaline, sulfametin, and sulfadimerazine were identified in foods by TLC with chloroform–isopropanol (4:1) and chloroform–methanol–hexane (16:3:1) and detection under UV light after dinitration [129]. Urine was screened for residues of sulfadiazine, sulfadimidine, sulfadimetoxine, and sulfaquinoxaline by TLC on silica gel with ethyl acetate–dichloromethane (4:1) after preconcentration with acetonitrile; detection was carried out by spraying with 0.02% fluorescamine in acetone and observation under 366 nm UV light [130]. Sulfadimidine, sulfadoxine, sulfadiazine, sulfadimethoxine, sulfathiazole, and sulfamerazine residues were screened in meat samples from retail outlets on silica gel layers with preadsorbent zone using a three-step development method with mixtures of ammonia in methanol and dichloromethane, detection by dipping in fluorescamine reagent, and semiquantitative determination by visual comparison of the zones against spiked samples [131]. Chloramphenicol, nitrofurazone, nitrofurantoin, furaltadone, furazolidone, sulfamethazine, sulfadimethoxine, sulfadoxine, and sulfamethoxypyridazine residues were screened in pork and beef muscle at 5–100 μg/kg by extraction with ethyl acetate, cleanup by silica gel SPE, and HPTLC on silica gel with preadsorbent zone using ethyl acetate–hexane (2:1) mobile phase; detection of nitrofurans was by spraying with pyridine and examination under 366 nm UV light and of sulfonamides and chloramphenicol by spraying with stannous chloride, NaOH, and fluorescamine solutions in turn [132].

3.15. Toxins

Reviews were published on the determination of ochratoxin A in animal and human tissues and fluids [133], fumonisin mycotoxins in maize and maize-based foods [134], and mycotoxins in foods [135] by TLC and other chromatographic methods.

The following papers were published on determination of aflatoxins B1, B2, G1, and G2: comparison of three methods for analysis of melon seeds showed that use of the TLC conditions of the official AOAC CB (Contaminants Branch) method, i.e., chloroform–acetone (22:3) mobile phase and UV detection at 360 nm, was superior to the BF (Best Food) and CB-RCS-Mod (modified CB method-Rapid Modification of the Cottonseed Method) in terms of less fluorescent interferences, better solvent efficiency, and lower detection levels [136]; slight modification of the CB method, with TLC plates submitted to double development followed by fluorescence densitometry, for application to cashews [137]; densitometric quantification in a variety of foods with a detection limit of 40 pg was accomplished on HPTLC silica gel with chloroform–xylene–acetone (6:3:1) or xylene–ethy1 acetate–acetic acid (6:3:1) mobile phase, after prewashing the plate by developing with diethyl ether and chloroform–xylene–acetone (6:3:1) in the opposite direction, and densitometry at 366 nm

[138]; quantification in poultry and pig feed and foodstuffs used in Colombia on silica gel with chloroform–acetone (9:1) with detection and densitometry at 366 nm [139]; and determination in dried red pepper samples by TLC and HPLC [140]. A TLC method developed for determination of aflatoxins B1 and M1, zearalenone, and ochratoxin A in bovine, swine, and fowl liver and kidney compared favorably with an AOAC reference method [141]. Parallel determination of two aflatoxin precursors, sterigmatocystin and *O*-methylsterigmatocystin, in milk with a detection limit of 1 μg/kg was described based on chloroform extraction, gel permeation chromatography cleanup, 2D silica gel TLC, and fluorodensitometry [142]. Nutmegs were screened for aflatoxins using TLC [143].

The quantitative determination of ochratoxin A (OA) in rice was performed by densitometry at 365 nm after HPTLC on silica gel developed with diethyl ether–methanol (49:1) in the reverse direction and then with toluene–ethyl acetate–formic acid (5:4:1) after cutting off the end of the plate closest to the zone origin and having the cut edge down in the tank, and finally with hexane–ethyl acetate–acetic acid (18:3:1) after cutting off a further 30 mm from the plate bottom; the method was validated and compared with the current AOAC method [144]. OA was determined in cereals and animal feed by acidified acetonitrile extraction, purification by combined pH-controlled liquid–liquid partition and strong anion-exchange SPE, and reversed-phase TLC; the detection limit was 8 ng/g and recovery 95% [145]. Two TLC methods for determination of OA in green coffee beans were compared, and both were found to be reliable for screening products imported by Greece and Lebanon (tolerance limit 20 ng/kg) [146]. A previously published method for determination of OA in corn, barley, and kidney was modified for application to parboiled rice with quantification by normal phase HPTLC; the method was validated over the range 0–198 μg/kg and showed advantages over a current AOAC method [147]. OA production ability by 148 *Aspergillus carbonarius* and *A. niger* isolates from mixed Middle Eastern and Australian sources was screened using coconut cream agar and TLC with fluorescence detection under longwave UV light [148].

An AOAC HPLC method for quantification of zearalenone was modified for application to maize with quantification by HPTLC, and the new method was validated over the range 10–320 μg/kg [149]. Zearalenone was extracted from corn flour according to a modified final-action AOAC method and determined by TLC with successive development with benzene–hexane (3:1) and toluene–ethyl acetate–formic acid (4:2:1) and detection as a bright red zone with bis-diazotized benzidine chromogenic agent; mean recoveries of 120–960 μg/kg ranged from 75 to 100% [150].

The occurrence of the mycotoxin patulin in apples with mouldy core was studied using TLC on silica gel with toluene–ethyl acetate–formic acid (5:4:1) and detection under 254 nm UV light [151]. Patulin was quantified in apples, apple concentrate, and apple juice by extraction with ethyl acetate, cleanup by partition with 1.5% sodium carbonate solution, separation on silica gel layers with toluene–ethy1 acetate–formic acid (6:3:1) mobile phase, and reflectance scanning densitometry at 275 nm or at 412 nm after detection with MBTH spray reagent; the detection limit was 30–50 ng/spot, RSD was 4%, and average recovery was 78% for concentrations between 50 and 150 ppb [152].

A method for fumonisin B in rice, validated over the range 0–16 μg/g, utilized strong anion-exchange SPE, normal-phase HPTLC, novel detection by dipping into a 0.16% acidic solution of *p*-anisaldehyde, and scanning fluorodensitometry [153]. The mushroom nephrotoxin orellanine was determined with a detection limit of 15 ng by TLC on cellulose plates developed with 1-butanol–acetic acid–water and fluorescence densitometry [154]. The analysis of fusarium toxins (deoxynivalenol, fumonisin B1, and zearalenone) in maize and wheat by TLC was compared to HPLC and enzyme-linked immunosorbent assay (ELISA); results were found to agree among methods, but TLC was least expensive [155]. Domoic acid was semiquantified in shellfish tissue with a sensitivity limit of 10 μg/g using extraction by single-step homogenization with 50% aq. ethanol, strong anion-exchange resin cleanup, silica gel TLC with butanol–acetic acid–water (3:1:1) mobile phase, detection by fluorescence quenching under 254 nm UV light, and confirmation by formation of a yellow zone by spraying with ninhydrin reagent [156].

3.16. Vitamins

The effects of microwave heating on the loss of vitamin B12 in foods were studied by TLC of OH-, CN-, Ado-, and Me-B12 on silica gel developed in the dark with 1-butanol–2-propanol–water (10:7:10) and quantification by densitometry at 527 nm [157]. Isomers of ascorbic acid and dehydroascorbic acid in food products were separated and identified by TLC on sodium borate impregnated silica gel, cellulose, and reversed-phase plates as well as unimpregnated silica gel and reversed-phase plates [158].

3.17. Miscellaneous compounds

Components from guarana oil (e.g., carvacrol, estragole, anethole, limonene, caryophyllene, and copaene) were qualitatively identified by TLC on silica gel with chloroform or benzene and detection by spraying with anisaldehyde and PMA solutions; for urinary metabolites of guarana, a mixture of 1-butanol–acetic acid–water (4:1:5) was used as mobile phase for ninhydrin-positive substances, and ethyl acetate–ethanol–ammonia (85:10:59) for amphetamines [159]. The ingredients in different cinnamons were characterized by TLC on silica gel developed with dichloromethane in a tank controlled at 40% relative humidity by a sulfuric acid–water solution placed inside, with detection by spraying with anisaldehyde–sulfuric acid, vanillin–sulfuric acid, and potassium hydroxide solutions; botanical differences were clearer, retention results more reproducible, and solvent costs and analysis time were less when TLC was compared to HPLC [160]. A qualitative TLC method on silica gel with hexane–acetone–decane–methanol (59:30:10:1) mobile phase was described to detect the animal drug ivermectin (see Section 4.4) as a fluorescent derivative in extracts of cattle dung with a limit of detection of 40 ng/g [161]. Propyl, octyl, and dodecyl gallates from olive oils and butters were identified by TLC and quantified by densitometry [162]. A new method for simultaneous quantification of hydroxamic acids in up to five extracts of wheat or rye was based on TLC densitometry [163]. Silica gel HPTLC, gradient elution, and UV scanning at 260–310 nm were applied to determine vanillin, vanillic acid, *p*-hydroxybenzaldehyde, and *p*-hydroxybenzoic acid in vanilla extracts, coumarin in cinnamon, and curcuminoids in tumeric [164,165]. α- and β-Asarone were determined in *Acorus calamus* and *A. europaeum* essential oils and alcoholic extracts used in foods and beverages by TLC and GC–MS [166]. The drugs nalidixic acid and flumequine were detected at 0.02–0.26 ppm in cultured fish and chicken by extraction with 0.1% *m*-phosphoric acid–methanol (3:7), C_{18} Sep-Pak cleanup, and silica gel TLC with sodium borohydride and hydrogen peroxide fluorogenic spray reagents [167].

4. Summaries of selected TLC and HPTLC methods for food and agricultural analyses

The following are brief summaries of selected applications of TLC for the determination of important compounds in food and agricultural samples. Classical ascending development of the layer with the mobile phase was carried out in an N-tank unless otherwise noted.

4.1. Ampicillin in milk and muscle of food-producing animals

Sample preparation: Protein precipitation with trichloroacetic acid, ampicillin hydrolysis with sodium hydroxide, reaction of the hydrolysis product with mercuric chloride in acidic medium to form a fluorescent compound that is extracted from the matrix with dichloromethane and concentrated by Sep-Pak silica gel SPE in a single step, evaporation of the combined cartridge eluents (hexane–ethyl acetate, 1:1, followed by 1:3), reconstitution with 100 μl of ethyl acetate, and spotting of 20 μl for TLC.

Layer: HPTLC silica gel 60 with preadsorbent zone.

Mobile phase: ethyl acetate.

Detection and quantification: determination at 4 and 50 μg/kg of milk and muscle by densitometry at 365 nm.

Ref.: [168].

4.2. Gentiopicroside in the roots of Gentia lutea L.

Sample preparation: roots were extracted with methanol–water (1:1) at 60°C.

Layer: silica gel 60F HPTLC.

Mobile phase: ethyl acetate–methyl ethyl ketone–acetic acid–water (5:3:1:1).

Detection: sulfuric acid reagent.

Quantification: densitometry at 530 nm.

Ref.: [169].

4.3. Heterocyclic oxygen compounds in citrus fruit essential oils

Sample preparation: commercial oils were analyzed without dilution or after 1:10 dilution with hexane.

Layer: silica gel 60F HPTLC.

Mobile phase: *n*-buty1 acetate–hexane (8:2) and chloroform–*n*-butyl acetate–hexane (9:1:15), overpressured development.

Detection: 254 and 366 nm UV light.

Ref.: [170].

4.4. Ivermectin residues in swine and cattle tissues

Sample preparation: tissue sample was extracted with acetonitrile followed by C_{18} SPE, derivatization with trifluoroacetic anhydride, and repeated SPE.

Layer: silica gel 60 with concentrating zone.

Mobile phase: ethyl acetate–chloroform (1:3).

Detection: ivermectin appears as two blue zones, one of which is much higher intensity than the other, under 366 nm UV light after air drying the plate and dipping into 10% paraffin in hexane; residues in meat, liver, and fat were detected at levels down to 5 μg/kg.

Ref.: [171].

4.5. Lipids (neutral lipids) in eggs

Sample preparation: extraction from egg yolk with chloroform–methanol (2:1).

Layer: channeled preadsorbent high performance silica gel.

Mobile phase: (1) light petroleum (b.p. 37.5–52°C)–diethyl ether–acetic acid (80:20:2) for determination of cholesterol, triacylglycerols, and free fatty acids; (2) *n*-hexane–light petroleum–diethyl ether–acetic acid (50:20:5:1) for cholesterol esters.

Detection: PMA (5% in ethanol) and heating at 110–120°C for 5–10 min.

Quantification: scanning densitometry at 700 nm.

Ref.: [172].

4.6. Monesin in feeds (screening method)

Sample preparation: extraction with methanol and purification by silica gel cartridge SPE.

Layer: aluminum-backed silica gel TLC sheets.

Mobile phase: ethyl acetate–dichloromethane–ammonia (34:6:1).

Detection: color development by spraying with vanillin reagent (3 g+0.5 ml sulfuric acid in 100 ml methanol) and heating at 120°C for 1–5 min; positive results are confirmed by a second color development by spraying with 0.5% *p*-anisaldehyde in 85 ml methanol+10 ml sulfuric acid+5 ml acetic acid and heating as above.

Ref.: [173].

4.7. Phenylurea herbicides in plants

Sample preparation: carrots, apples, asparagus, and wheat were extracted with acetone and extracts purified by silica SPE.

Layer: silica gel 60F HPTLC.

Mobile phase: AMD with a 25 cycle gradient composed of acetonitrile, dichloromethane, acetic acid, toluene, and hexane in varying proportions.

Quantification: densitometry at 245 nm; 0.01 ppm limit of detection.

Ref.: [174].

4.8. Pigments (bixin, lycopene, canthaxanthin, and β-apo-8′-carotenal) in products derived from red pepper

Sample preparation: acetone extraction, ether partitioning, and saponification.

Layer: silica gel F.

Mobile phase: hexane–acetone (10:9), dichloromethane–diethy1 ether (9:l), light petroleum (b.p. 65–95°C)–benzene (1:1), and light petroleum (b.p. 40–60°C).

Detection: natural color.

Quantification: spectroscopy after elution.
Ref.: [175].

4.9. Polyphosphates in seafood

Sample preparation: fish were homogenized, mixed with water (1:2) for 1 min, and the filtrate collected over No. 4 filter paper.
Layer: cellulose.
Mobile phase: 2-propanol–1-propanol–trichloroacetic acid–ammonium hydroxide–water (200 ml:175 ml:25 g:1 ml:125 ml).
Detection: molybdenum blue spray reagent followed by sodium pyrosulfite–sodium sulfite–methylaminophenol reagent.
Ref.: [176].

4.10. Quinine in tonic water

Sample preparation:degassed tonic waters were spotted directly.
Layer: channeled preadsorbent high-performance silica gel.
Mobile phase: toluene–acetone–diethyl ether–ammonium hydroxide–methanol (12:18:6:1.5:1).
Detection: natural fluorescence.
Quantification: fluorescence densitometry, 350 nm excitation wavelength.
Ref.: [177].

4.11. Stilbene phytoalexins in peanuts

Sample preparation: extraction by blending with acetone–acetonitrile (9:1) and cleanup on an alumina–C_{18} (1:1) column eluted with acetonitrile–water (9:1).
Layer: silica gel 60.
Mobile phase: hexane–2-propanol–water–acetic acid (80:18:1:1).
Detection: under 366 nm UV light and by spraying with aq. 0.5% fast blue B solution followed, if needed, by spraying with potassium carbonate solution or placing the plate for 1–2 s in a TLC chamber saturated with ammonia; qualitative identification by TLC and quantification by HPLC at 100–300 ppb.
Ref.: [178].

4.12. Sugars in beverages

Sample preparation: sodas and iced teas were diluted with ethanol–water (7:3) and spotted directly.
Layer: HPTLC silica gel 60 with 19 lanes and preadsorbent zone, impregnated by spraying with 0.10 *M* sodium hydrogen sulfite and pH 4.8 citrate buffer solutions.
Mobile phase: three 7 cm developments with acetonitrile–water (85:15).
Detection: as purple zones by spraying with 1-naphthol (5 g)–ethanol (160 ml)–sulfuric acid (20 ml)–water (13 ml) solution and heating for 5–10 min at 110°C.
Quantification: densitometry at 515 nm; recovery of fructose, glucose, and sucrose averaged 94% and the RSD was 2.5%.
Ref.: [179].

4.13. Thiamphenicol in bovine and human plasma

Sample preparation: plasma was extracted with ethyl acetate and the extract cleaned up by 1iquid–liquid partitioning at controlled pH.
Layer: amino, cyano, and silica gel layers.
Mobile phase: ethyl acetate–methanol (5:1), methanol–water (1:1), and ethyl acetate–acetic acid (100:1), respectively.
Detection and quantification: densitometry at 230 nm with florphenicol internal standard.
Ref.: [180].

4.14. Tinuvin 622 (polyolefin stabilizer) in extracts derived from polyolefinic foodstuffs-packaging materials

Sample preparation: food-simulating extractant liquids (distilled water, aqueous acetic acid, aqueous ethanol, and sunflower oil) were analyzed.
Layer: cellulose (prewashed with the mobile phase).
Mobile phase: 2-propanol–25% acetic acid–toluene (10:10:1).
Detection: iodine vapor.
Semiquantitative evaluation: comparison of the sizes of sample and standard zones.
Ref.: [181].

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